Data Evaluation Report on the terrestrial field dissipation of pinoxaden

PMRA Submission Number {.....} EPA MRID Number 46203023

Data Requirement: PMRA Data Code:
EPA DP Barcode: D300622
OECD Data Point:
EPA Guideline: 164-1

Test material: NOA-407855

End Use Product name: NOA-407855 120EC **Concentration of a.i.:** 12%
Formulation type: Emulsifiable concentrate

Active ingredient
Common name: Pinoxaden.
Chemical name
IUPAC: 8-(2,6-Diethyl-p-tolyl)-1,2,4,5-tetrahydro-7-oxo-7H-pyrazolo[1,2-d][1,4,5]-oxadiazepin-9-yl 2,2-dimethylpropionate.
CAS name: 8-(2,6-Diethyl-4-methylphenyl)-1,2,4,5-tetrahydro-7-oxo-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-9-yl 2,2-dimethylpropanoate.
8-(2,6-Diethyl-4-methylphenyl)-1,2,4,5-tetrahydro-7-oxo-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-9-yl ester 2,2-dimethylpropanoic acid.
CAS No: 243973-20-8.
Synonyms: NOA-407855.
SMILES string: O=C1C(=C(N2N1CCOCC2)OC(=O)C(C)(C)C)c1c(cc(cc1CC)C)CC.

Primary Reviewer: Dan Hunt **Signature:**
Dynamac Corporation **Date:**

QC Reviewer: Joan Harlin **Signature:**
Dynamac Corporation **Date:**

Secondary Reviewer: Ibrahim Abdel-Saheb **Signature:**
Environmental Risk Branch II
Environmental Fate and Effects Division (7507C) **Date:** 1/10/05

Company Code:
Active Code:
Use Site Category:
EPA PC Code: 147500

CITATION: Speth, R.M. 2004. Terrestrial field dissipation of NOA-407855 120EC on bare soil and spring wheat in North Dakota. Unpublished study performed by Waterborne Environmental, Inc., Leesburg, VA (field management), AGVISE Research, Inc., Northwood, ND (field research facility), Agvise Laboratories, Northwood, ND (soil characterization), Syngenta Crop Protection,

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Inc., Greensboro, NC (sample processing), and Environ-Test Laboratories, Edmonton, Alberta, Canada (analytical laboratory), and sponsored and sponsored and submitted by Syngenta Crop Protection, Inc., Greensboro, NC. Syngenta Number: 32-01. Study initiation June 26, 2001 and completion January 19, 2004. Final report issued January 19, 2004.

EXECUTIVE SUMMARY:

Soil dissipation/accumulation of pinoxaden (8-(2,6-diethyl-p-tolyl)-1,2,4,5-tetrahydro-7-oxo-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-9-yl 2,2-dimethylpropionate; NOA-407855) under US field conditions was conducted in a bare ground plot and a plot cropped with spring wheat at one site in Grand Forks County, North Dakota (Ecoregion not reported). The experiment was carried out in accordance with the USEPA Pesticide Assessment Guidelines Subdivision N, §164-1 and in compliance with the USEPA FIFRA (40 CFR, Part 160) GLP standard. Pinoxaden was broadcast once at a target application rate of 0.077 kg a.i./ha (0.0687 lb a.i./A) onto two plots (one bare ground and one cropped with spring wheat) of loam soil (0-30 cm) that were each divided into three replicate plots measuring approximately 9 x 36 m. Pinoxaden was applied with the safener, cloquintocet-mexyl (CGA-185072). The spring wheat was at the 6-leaf growth stage at the time of test substance application. The target application rate corresponds to 110% of the current maximum annual application rate for control of grass weeds in small grain crops. Rainfall was supplemented with irrigation to reach 107% of the 30-year average rainfall. The two treated plots were located approximately 1.5 m apart from each other, and the control plots were located approximately 15 m away from the treated plots.

The application rate was verified using petri dish tops (15-cm diameter) containing filter paper that were placed in the bare ground and cropped plots prior to application. The mean recoveries of pinoxaden from the filter papers placed in the treated bare ground and cropped plots were 106% and 104% of the theoretical application rate, respectively. Field spiking was done by fortifying control soil samples with pinoxaden and its transformation products NOA-407854 and NOA-447204 at a concentration of 10 ppb. Mean recoveries of pinoxaden, NOA-407854, and NOA-447204 from the field spiked soil samples ranged from 89-93%, 80-81%, and 75-81%, respectively. Samples were stored frozen for up to 196 days prior to analysis.

Soil samples were collected from the treated bare ground and cropped plots at -3, 0, 0.5, 1, 3, 7, 14, 21, 35, 45, 59, 88, 125, 305, 363, 454, and 494 days following the test application to a depth of 0-120 cm (except day-0 and day-0.5 samples which were collected to a depth of 15 cm). Soil samples were analyzed for pinoxaden and its transformation products NOA-407854 (7H-pyrazolo[1,2-d][1,4,5]oxadiazepine-7,9(8H)-dione, 8-(2,6-diethyl-4-methylphenyl)tetrahydro-) and NOA-447204 (7H-pyrazolo[1,2-d][1,4,5]oxadiazepine-7,9(8H)-dione, 8-(2,6-diethyl-4-methylphenyl)tetrahydro-8-hydroxy-), and the safener, cloquintocet-mexyl and its transformation product CGA-153433. Soil samples (10 g) were extracted by sonicating with 60 mL of acetone:water (60:40, v:v) containing pH 3 citrate buffer for 15 minutes and shaking for 45 minutes. An aliquot of the extract was passed through a 2-gram aminopropyl SPE column and a Nexus Abselut SPE column, and analyzed for pinoxaden, NOA-407854, NOA-447204, and cloquintocet-mexyl by LC/MS/MS. The remaining extract was extracted twice by shaking with dichloromethane and cleaned-up on a Varian bond-elute C18 SPE column prior to analysis for CGA-153433 by LC/MS/MS. The LOQ for all analytes in soil was 0.5 ppb. Soil samples were stored frozen for up to 442 days (bare ground plot) and 449 days (cropped plot) prior to analysis (does not include re-analyses).

In the bare ground plot, the measured zero-time concentration in the 0-15 cm soil depth was 22 ppb, which is 45% of the applied rate (reviewer-calculated based on a theoretical day-0 concentration of 0.049 mg a.i./kg in the 0-15 cm soil depth). The day-0 concentration of pinoxaden + NOA-407854 (after converting to parent equivalents) was 73% of the applied rate (reviewer-calculated). Following day 0, pinoxaden decreased to 7.5 ppb by 3 days and was last detected above the LOQ at 1.3 ppb at 14 days posttreatment. **NOA-407854** was initially detected in the 0-15 cm soil depth at 11 ppb immediately following the application (which is 28.4% of the applied pinoxaden, after converting to parent equivalents). NOA-407854 increased to a maximum concentration of 14 ppb by 0.5 days (which is 36.2% of the applied pinoxaden, after converting to parent equivalents), then decreased to 6.1 ppb by 3 days, and was last detected above the LOQ at 0.74 ppb at 21 days posttreatment. The registrant-calculated half-life value for NOA-407854 was 3.3 days, calculated using non-linear regression. **NOA-447204** was initially detected in the 0-15 cm soil depth at 0.54 ppb at 0.5 days, increased to a maximum concentration of 4.2 ppb by 7 days (which is 10.5% of the applied pinoxaden, after converting to parent equivalents), then decreased to 2.7 ppb by 125 days, and was 0.58 ppb at 494 days posttreatment. Pinoxaden and its transformation products were not detected above the LOQ in soil below the 0-15 cm depth.

In the cropped plot, the maximum measured concentration in the 0-15 cm soil depth was 8.5 ppb at 0.5 days, which is 18% of the applied rate (reviewer-calculated based on a theoretical day-0 concentration of 0.048 mg a.i./kg in the 0-15 cm soil depth). The maximum concentration of pinoxaden + NOA-407854 (after converting to parent equivalents) was 27% of the applied rate at 0.5 days posttreatment (reviewer-calculated). Following 0.5 days, pinoxaden decreased to 4.1 ppb by 3 days and was last detected above the LOQ at 0.56 ppb at 14 days posttreatment. **NOA-407854** was detected in the 0-15 cm soil depth at a maximum concentration of 4.0 ppb immediately following the application (which is 10.5% of the applied pinoxaden, after converting to parent equivalents), and decreased to a final concentration of 1.3 ppb by 7 days posttreatment. The registrant-calculated half-life value for NOA-407854 was 5.8 days, calculated using non-linear regression. **NOA-447204** was initially detected in the 0-15 cm soil depth at 1.0 ppb at 3 days, increased to a maximum concentration of 4.1 ppb by 35-45 days (which is 10.4% of the applied pinoxaden, after converting to parent equivalents), then decreased to 0.73 ppb by 494 days posttreatment. Pinoxaden and its transformation products were not detected above the LOQ in soil below the 0-15 cm depth.

Under field conditions at the test site in the bare ground plot, the reviewer-calculated half-life of pinoxaden in soil under terrestrial field conditions was 3 days ($r^2 = 0.90$), calculated using linear regression analysis performed on a plot of ln-transformed concentrations vs. time and the equation $t_{½} = ln\ 2 / k$, where k is the rate constant. A DT90 value was not calculated. At the end of the 494-day study period, the total carryover of residues of pinoxaden and its transformation products was 1.4% of the applied, based on the target application rate.

Under field conditions at the test site in the cropped plot, the reviewer-calculated half-life of pinoxaden in soil under terrestrial field conditions was 4 days ($r^2 = 0.81$), calculated using linear regression analysis performed on a plot of ln-transformed concentrations vs. time and the equation $t_{½} = ln\ 2 / k$, where k is the rate constant. A DT90 value was not calculated. At the end of the 494-

day study period, the total carryover of residues of pinoxaden and its transformation products was 1.9% of the applied, based on the target application rate.

The major route of dissipation of pinoxaden under terrestrial field conditions was transformation.

RESULTS SYNOPSIS

Location/soil type: Near Northwood, North Dakota/loam-clay loam (0-90 cm).
Half-lives (reviewer-calculated):

Bare ground plot: 3 days ($r^2 = 0.90$).

Cropped plot: 4 days ($r^2 = 0.81$).

Major transformation products detected:

NOA-407854 (7H-pyrazolo[1,2-d][1,4,5]oxadiazepine-7,9(8H)-dione, 8-(2,6-diethyl-4-methylphenyl)tetrahydro-).

NOA-447204 (7H-pyrazolo[1,2-d][1,4,5]oxadiazepine-7,9(8H)-dione, 8-(2,6-diethyl-4-methylphenyl)tetrahydro-8-hydroxy-).

Dissipation routes: Transformation.

Study Acceptability: This study is classified **supplemental** and does not satisfy the guideline requirement for a terrestrial field dissipation study because the concentration of pinoxaden in the field samples at the time of analysis may not accurately represent the concentration in the field at the time of sampling, since it is possible that pinoxaden degraded in the samples during their prolonged frozen storage prior to analysis. No additional terrestrial field dissipation study is required.

I. MATERIALS AND METHODS

GUIDELINE FOLLOWED: The study was conducted according to USEPA Pesticide Assessment Guidelines Subdivision N, 164-1. One significant deviation from EPA Subdivision N, 164-1 noted was:

> The concentration of pinoxaden in the field samples at the time of analysis may not accurately represent the concentration in the field at the time of sampling, since it is possible that pinoxaden degraded in the samples during their prolonged frozen storage prior to analysis. This does not affect the validity of the study.

COMPLIANCE: The study was conducted in compliance with USEPA FIFRA (40 CRF Part 160) Good Laboratory Practice standards. Signed and dated GLP Compliance, Quality Assurance, and Data Confidentiality statements were provided.

A. MATERIALS:

1. Test Material Pinoxaden (NOA-407855).

Chemical Structure of the active ingredient(s): See DER Attachment 1.

Description: Emulsifiable concentrate.

Storage conditions of test chemicals: <-10°C (Appendix 2, Figure 1).

Physico-chemical properties of pinoxaden.

Parameter	Values	Comments
Water solubility	Not reported	
Vapour pressure/volatility	Not reported	
UV absorption	Not reported	
pKa	Not reported	
K_{ow}/log K_{ow}	Not reported	
Stability of Compound at room temperature	Not reported	

2. Test site: The test site was located approximately two miles west of Northwood, North Dakota (in Grand Forks County), an area representative geographically and climatically of the Northern Great Plains region of the United States where spring wheat is principally grown (Appendix 1, Figures 1-2). The treated and control plots had been fallow in 2000 and had been used to grow dry beans in 1999, soybeans in 1998, and sugarbeets in 1997 (Appendix 1, Table 2). Sonolan, Basagran, Assure II, Prowl, Glean, and Ally had been applied during the four years prior to the application of the test substance.

Table 1: Geographic location, site description and climatic data at the study site.

Details		Test site
Geographic coordinates	Latitude	Not reported
	Longitude	Not reported
	Province/State	North Dakota
	Country	US
	Ecoregion	Not reported
Slope Gradient		<1.0% (est.)

Details	Test site
Depth to ground water (m)	>1.2 m (est.)
Distance from weather station used for climatic measurements	Daily precipitation data were obtained from one of three locations during the study: a rain gauge near the test site or weather stations located 400 ft and 11 miles from the test site. Soil temperatures were measured on-site and air temperatures and evapotranspiration were recorded 400 ft from the test site.
Indicate whether the meterological conditions before starting or during the study were within 30 year normal levels (Yes/No). If no, provide details.	Total water input (rainfall plus irrigation) during the study period (494 days) was 31.18 inches or 107% of the 30-year average rainfall.

Data were obtained from pp. 17, 19 and 25, and Appendix 1, Table 7, p. 99 of the study report.

Table 2: Site usage and management history for the previous three years.

Use	Year	Test site
Crops grown	Previous year	Fallow
	2 years previous	Dry beans
	3 years previous	Soy beans
Pesticides used	Previous year	None
	2 years previous	Sonalan and Basagran.
	3 years previous	Assure II, Sonalan, and Basagran.
Fertilizers used	Previous year	Not reported
	2 years previous	Not reported
	3 years previous	Not reported
Cultivation methods, if provided (eg., Tillage)	Previous year	Not reported
	2 years previous	Not reported
	3 years previous	Not reported

Data were obtained from Appendix 1, Table 2, p. 94 of the study report.

3. Soils:

Table 3: Properties of the soil from the bare ground plot (Gardena silt loam).

Property	Depth (cm)							
	0-15	**15-30**	**30-45**	**45-60**	**60-75**	**75-90**	**90-105**	**105-120**
Textural classification	L	L	CL	CL	CL	L	SL	SL
% sand	32	30	30	24	32	32	24	28
% silt	46	48	42	40	36	44	54	52
% clay	22	22	28	36	32	24	22	20
pH	7.3	7.8	8.3	8.5	8.6	8.6	8.5	8.4
Total organic matter (%)	5.0	4.1	1.9	1.3	0.9	0.6	0.5	0.4
CEC (meq/100 g)	23.8	28.1	27.5	25.9	25.1	23.8	24.3	23.6
Bulk density (g/cm^3)	1.04	1.11	1.09	1.06	1.06	1.09	1.08	1.07
Moisture at 1/3 atm (%)	32.9	37.0	34.5	33.7	33.5	29.9	33.2	32.2
Taxonomic classification (e.g., ferro-humic podzol)	Coarse-silty, mixed, superactive, frigid Pachic Hapludolls							
Soil mapping unit	Not available.							

Data were obtained from pp. 17-18 and Appendix 1, Table 3, p. 95 of the study report. L = Loam, CL = Clay loam, SL = Silt loam. The taxonomic classification was obtained from the NRCS for the Gardena soil series.

Table 4: Properties of the soil from the cropped plot (Gardena silt loam).

Property	Depth (cm)							
	0-15	**15-30**	**30-45**	**45-60**	**60-75**	**75-90**	**90-105**	**105-120**
Textural classification	L	L	L	CL	CL	L	L	L
% sand	42	40	40	32	32	30	30	46
% silt	40	44	40	40	40	48	50	40
% clay	18	16	20	28	28	22	20	14
pH	7.1	7.8	8.2	8.5	8.7	8.7	8.6	8.5
Total organic matter (%)	4.7	3.9	2.1	1.1	0.8	0.5	0.5	0.4
CEC (meq/100 g)	22.9	24.3	25.5	23.2	23.1	21.6	23.0	21.4
Bulk density (g/cm^3)	1.06	1.01	1.10	1.11	1.12	1.09	1.10	1.08
Moisture at 1/3 atm (%)	34.0	31.0	28.8	29.4	28.5	27.5	31.2	27.9
Taxonomic classification (e.g., ferro-humic podzol)	Coarse-silty, mixed, superactive, frigid Pachic Hapludolls							
Soil mapping unit	Not available.							

Data were obtained from pp. 17-18 and Appendix 1, Table 3, p. 95 of the study report. L = Loam, CL = Clay loam. The taxonomic classification was obtained from the NRCS for the Gardena soil series.

B. EXPERIMENTAL DESIGN:

1. Experimental design:

Table 5: Experimental design.

Details		Bare ground plot	Cropped plot
Duration of study		494 days	494 days
Uncropped (bare) or cropped		Bare	Cropped
Control used (Yes/No)		Yes	Yes
No. of replications	Controls	One	One
	Treatments	Three	Three
Plot size (L x W m)	Control	18.3 x 16.7 m	18.3 x 16.7 m
	Treatment	35.6 x 9.1 m/replicate plot	35.6 x 9.1 m/replicate plot
Distance between control plot and treated plot		15.2 m to the nearest treated plot	15.2 m to the nearest treated plot
Distance between treated plots		1.5 m between replicate plots	1.5 m between replicate plots
Application rate(s) used (g a.i/ha)		77 g a.i./ha	77 g a.i./ha
Was the maximum label rate per ha used in study? (Yes/No)		The application rate was 110% of the current maximum annual rate for small grain crops.	The application rate was 110% of the current maximum annual rate for small grain crops.
Number of applications		One	One
Application Date(s) (dd mm yyyy)		29/06/2001	29/06/2001
For multiple applications, application rate at Day 0 and at each application time (mg a.i./kg soil)[1]		0.049 mg a.i./kg soil	0.048 mg a.i./kg soil
Application method (eg., spraying, broadcast etc.)		Broadcast	Broadcast
Type of spray equipment, if used		Tractor-mounted boom sprayer with 14 XR8004VS flat fan nozzles. The spray height was 18 inches above the soil.	Tractor-mounted boom sprayer with 14 XR8004VS flat fan nozzles. The spray height was 18 inches above the soil.
Total volume of spray solution applied/plot **OR** total amount broadcasted/plot[2]		32,077 mL	32,077 mL
Identification and volume of carrier (e.g., water), if used		Water	Water

Details		Bare ground plot	Cropped plot
Name and concentration of co-solvents, adjuvants and/or surfactants, if used		Merge™ (cloquintocet-mexyl; CGA-185072) at 0.70%.	Merge™ (cloquintocet-mexyl; CGA-185072) at 0.70%.
Indicate whether the following monthly reports were submitted: Precipitation Average minimum and maximum air temperature Average minimum and maximum soil temperature Average annual frost-free periods		 Yes Yes Mean only (3-inch depth) No	 Yes Yes Mean only (3-inch depth) No
Indicate whether the Pan evaporation data were submitted		No, evapotranspiration data were reported.	No, evapotranspiration data were reported.
Meteorological conditions during application	Cloud cover	5%	20%
	Temperature (°C)	24.4°C	26.7 °C
	Relative humidity	82%	79%
	Wind speed	0-4 mph, S	0-6 mph, SW
	Sunlight (hr)	Not reported	Not reported
Pesticides used during study: name of product/a.i concentration: amount applied: application method: name of product/a.i concentration: amount applied: application method:		 Roundup Ultra Three times at 1 qt/A Not reported Roundup Ultra Max Two times at 1 qt/A Not reported	 2,4-D Once at 1 pt/A Not reported Roundup Ultra Max Two times at 1 qt/A Not reported
Supplemental irrigation used (Yes/No) If yes, provide the following details: No. of irrigation: Interval between irrigation: Amount of water added each time: Method of irrigation:		Yes 9 1 day- 8 months 0.16-1.07 inches Overhead sprinkler	Yes 9 1 day- 8 months 0.16-1.07 inches Overhead sprinkler
Indicate whether water received through rainfall + irrigation equals the 30 year average rainfall (Yes/No)		Yes	Yes
Were the application concentrations verified? (Briefly describe in Section 2, if used)		Yes	Yes
Were field spikes used? (Briefly describe in Section 3, if used)		Yes	Yes
Good agricultural practices followed (Yes or No)		Not reported	Not reported

Details	Bare ground plot	Cropped plot
Indicate if any abnormal climatic events occurred during the study (eg., drought, heavy rainfall, flooding, storm etc.)	None	None
If cropped plots are used, provide the following details: Plant - Common name/variety: Details of planting: Crop maintenance (eg., fertilizers used):	N/A	 Spring wheat/Russ Planted on June 1, 2001 at a seeding rate of 120 lb/A. 46-0-0 fertilizer was applied on May 22, 2001. The wheat crop was harvested on September 24, 2001.
Volatilization included in the study (Yes/No) (if included, describe in Section 4)	No	No
Leaching included in the study (Yes/No) (if included, describe in Section 5)	Yes	Yes
Run off included in the study (Yes/No) (if included, describe in Section 6)	No	No

Data were obtained from pp. 17-21 and Appendix 1, Tables 6-10, pp. 98-102, Figures 3-5, pp. 112-114 and pp. 117-134 of the study report.

[1] The application rate at day 0 was calculated by the reviewer based on the target application rate of 77 g a.i./ha and using a site bulk density of 1.04 g/cm^3 for the bare ground plot and 1.06 g/cm^3 for the cropped plot, and a soil depth of 15 cm for both plots. The registrant calculated a theoretical value of 34 ppb for the bare ground and cropped plots, based on a bulk density of 1.50 g/cm^3 (p. 29).

[2] Reviewer-calculated based on the number of tractor passes (five passes of 90 ft = 450 ft total pass), the target tractor speed of 4 ft/sec, and the sprayer output data (285.13 ml/sec; p. 20 and Appendix 1, Table 8, p. 100).

2. Application Verification: Fifteen petri dish tops (15-cm diameter) containing filter paper (15-cm diameter) were randomly placed in the bare ground and cropped plots prior to application. After application, the petri dish tops were collected and shipped frozen to the analytical laboratory.

The concentration of pinoxaden in the spray tank solutions was verified by collecting five 10-mL spray solution samples from the tank mix (three samples before application and two samples after application).

3. Field Spiking: Field spikes were prepared in triplicate for pinoxaden and its transformation products NOA-407854 and NOA-447204 (Appendix 1). Control soil samples (10 g) collected from the 0-15 cm soil layer from the bare ground control plot were fortified in triplicate with each analyte at a concentration of 10 ppb on September 26, 2002 (454 days posttreatment). Samples were analyzed after 196 days of storage.

4. Volatilization: Volatilization was not measured.

5. Leaching: Fifteen cores were collected from the treated bare ground plot and twelve cores were collected from the treated cropped plot at -3, 0, 0.5, 1, 3, 7, 14, 21, 35, 45, 59, 88, 125, 305, 363, 454, and 494 days following the test application to a depth of 120 cm (except day-0 and day-0.5 samples which were collected to a depth of 15 cm) to determine the mobility of the test substance in the soil profile. Five cores were collected from the two control plots at each sampling interval with the exception of day 0.5.

6. Run off: Run off was not studied.

7. Supplementary Study: A soil freezer storage stability study was conducted as a separate study and submitted as MRID 46203022. In the storage stability study, soil samples from the terrestrial field dissipation study site were fortified with pinoxaden and its transformation products NOA-407854 and NOA-447204 to achieve a concentration of 5 ppb for each analyte. Samples were analyzed following up to a maximum of 12 months of storage, with additional samples scheduled for analysis following up to 18 months of storage.

8. Sampling:

Table 6: Soil sampling.

Details	Bare ground plot	Cropped plot
Method of sampling (random or systematic)	Random	Random
Sampling intervals	-3, 0, 0.5, 1, 3, 7, 14, 21, 35, 45, 59, 88, 125, 305, 363, 454, and 494 days following application.	-3, 0, 0.5, 1, 3, 7, 14, 21, 35, 45, 59, 88, 125, 305, 363, 454, and 494 days following application.
Method of soil collection (eg., cores)	Cores	Cores
Sampling depth	120 cm (48 inches)[1]	120 cm (48 inches)[1]
Number of cores collected per plot	15/treated plot (5 replicates from each of the 3 subplots) and 5/control plot.	12/treated plot (4 replicates from each of the 3 subplots) and 5/control plot.
Number of segments per core	Eight	Eight
Length of soil segments	15 cm	15 cm
Core diameter (Provide details if more than one width)	5.4 cm (2 1/8 inches) for the 0-15 cm depth samples and 4.1 cm (1.6 inches) for the 15-120 cm depth samples.	5.4 cm (2 1/8 inches) for the 0-15 cm depth samples and 4.1 cm (1.6 inches) for the 15-120 cm depth samples.
Method of sample processing, if any	Soil samples were composited by subplot and depth to form three samples for each interval/depth combination, and then mixed in a Hobart™ foodchopper with dry ice.	Soil samples were composited by subplot and depth to form three samples for each interval/depth combination, and then mixed in a Hobart™ foodchopper with dry ice.

Details	Bare ground plot	Cropped plot
Storage conditions	Frozen	Frozen
Storage length[2]	Up to 442 days.	Up to 449 days.

Data were obtained from pp. 21-24 and Appendix 1, Table 10, p. 102, and Appendix 2, p. 206 and Table 7, pp. 231-236, and Table 14, pp. 256-262 of the study report.

[1] Excludes day-0 and day-0.5 samples which were collected to a depth of 15 cm (6 inches).

[2] Excludes re-analysis samples. Maximum storage length of samples was 745 days for the bare ground plot and 761 days for the cropped plot.

9. Analytical Procedures: The analytical method used for determining pinoxaden and its transformation products NOA-407854 (7H-pyrazolo[1,2-d][1,4,5]oxadiazepine-7,9(8H)-dione, 8-(2,6-diethyl-4-methylphenyl)tetrahydro-) and NOA-447204 (7H-pyrazolo[1,2-d][1,4,5]oxadiazepine-7,9(8H)-dione, 8-(2,6-diethyl-4-methylphenyl)tetrahydro-8-hydroxy-) and the safener, cloquintocet-mexyl in soil was Syngenta method No. 35-01, and the analytical method used for the determination of the cloquintocet-mexyl transformation product CGA-153433 (acetic acid, [(5-chloro-8-quinolinyl)oxy-) in soil was Enviro-Test method 262a (Appendix 2, Figure 1). The LOQ for all analytes in soil was 0.50 ppb.

Analytical method for the determination of pinoxaden, NOA-407854, NOA-447204 and cloquintocet-mexyl. Soil samples (10 g) were extracted by sonicating with 60 mL of acetone:water (60:40, v:v) containing pH 3 citrate buffer for 15 minutes and shaking for 45 minutes (Appendix 2; Figure 2). The samples were centrifuged and the extraction was repeated with 20 mL of extraction solution. The volume of the extract was adjusted to 80 mL with acetone and a 72-mL aliquot of the extract was transferred to a 2-gram aminopropyl SPE column (the remaining 8-mL aliquot was held for analysis of CGA-153433). The eluate was collected and the acetone was removed by rotary evaporation. After the addition of 5 mL of brine solution and 100 µL of acetic acid, the extract was transferred to a 1-gram Nexus Abselut SPE column. Analytes were eluted from the SPE cartridge using 2% formic acid in methanol, and the eluent was brought up to 50 mL with water and analyzed by HPLC (Waters Symmetry® Shield RP8 column, 100 x 3 mm) using MS/MS detection (Atmospheric Pressure Ionization Tandem Mass Spectroscopy; Appendix 2, Figure 4). The mobile phase conditions for the separation consisted of 0.1% acetic acid in water:acetonitrile (90:10 to 10:90 to 90:10, v:v). The retention times were 7:35 minutes for NOA-447204 and NOA-407854, 8:40 minutes for pinoxaden, and 9:45 minutes for cloquintocet-mexyl.

Analytical method for the determination of CGA-153433. An 8-mL aliquot of the above extract was extracted twice by shaking with 3 mL of dichloromethane, and the extracts were combined and concentrated (Appendix 2; Figure 3). The residue was re-dissolved in 2 mL of 2% formic acid:methanol, then adjusted to 10 mL with water and loaded onto a Varian bond-elute C18 SPE column. CGA-153433 was eluted from the column with 5 mL of methanol:water containing 1% formic acid (1:1, v:v) and analyzed by LC/MS/MS (Keystone Betasil C18 column, 100 x 4.6 mm) with a mobile phase gradient of 0.1% acetic acid in water:acetonitrile (95:5 to 35:65 to 95:5, v:v; Appendix 2, Figure 5). The retention time for CGA-153433 was 6:55 minutes.

The analytical purities of the reference standards were 99.6% for pinoxaden, 97.7% for NOA-407854, 94.5-97.8% for NOA-447204, 96.2% for cloquintocet-mexyl, and 99.8% for CGA-153433 (Appendix 2, Figure 1).

A method validation study was conducted prior to the analysis of the test samples using 0-15 cm depth control soil samples that were fortified in triplicate with pinoxaden, NOA-407854, and NOA-447204 at concentrations of 0.50, 5.0, and 50 ppb. Mean recoveries (across all fortification levels) were 103 ± 9.8% for pinoxaden, 81 ± 11.3% for NOA-407854, and 89 ± 14.4% for NOA-447204 (Appendix 2, Table 1). Recoveries did not differ significantly for the three fortification levels.

II. RESULTS AND DISCUSSION

1. APPLICATION MONITORS: The mean recoveries of pinoxaden from the filter papers placed in the treated bare ground and cropped plots were 106% and 104% of the theoretical application rate, respectively (Table 2). The recovery of pinoxaden includes contribution from NOA-407854 when present.

Mean concentrations of pinoxaden from the tank mix samples collected prior to and following the application were 103% and 99% of the theoretical concentration, respectively, for the bare ground plot; corresponding concentrations for the cropped plot were 83% and 77% of theoretical (Table 1).

2. RECOVERY FROM FIELD SPIKES: Mean recoveries of pinoxaden, NOA-407854, and NOA-447204 from the field spiked soil samples ranged from 89-93%, 80-81%, and 75-81%, respectively, and were approximately equal to the average procedural recoveries (Table 5).

3. MASS ACCOUNTING: A mass balance was not determined.

Table 7. Mean concentration of pinoxaden and cloquintocet-mexyl residues expressed as ppb soil in the bare ground plot (n=3).

Compound	Soil depth (cm)	Sampling times (days)															
		0	0.5	1	3	7	14	21	35	45	59	88	125	305	363	454	494
Pinoxaden (NOA-407855)	0-15	22	19	19	7.5	2.9	1.3	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	15-30	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	30-45	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
NOA-407854	0-15	11	14	7.8	6.1	2.9	1.0	0.74	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	15-30	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	30-45	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
NOA-447204	0-15	<0.50	0.54	0.73	2.1	4.2	4.1	4.1	4.1	3.1	2.7	2.7	2.7	1.2	0.79	0.52	0.58
	15-30	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	30-45	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
Cloquintocet-mexyl (CGA-185072)	0-15	7.5	8.7	6.8	4.6	3.8	2.6	1.1	0.53	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	15-30	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	30-45	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
CGA-153433	0-15	<0.50	<0.50	<0.50	0.68	0.90	0.73	1.6	1.3	1.0	0.88	0.88	0.69	1.3	0.77	<0.50	0.72
	15-30	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	30-45	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50

Data were obtained from Tables 4a-4c, pp. 41-43 of the study report. Mean values are averages of three replicates. The method LOQ was 0.5 ppb for each analyte in soil. Total extractable and non-extractable residues and total recovery were not determined.
NS = no sample.

Table 8. Mean concentration of pinoxaden and cloquintocet-mexyl residues expressed as ppb soil in the cropped plot (n=3).

Compound	Soil depth (cm)	Sampling times (days)															
		0	0.5	1	3	7	14	21	35	45	59	88	125	305	363	454	494
Pinoxaden (NOA-407855)	0-15	**7.8**	**8.5**	**7.4**	**4.1**	**1.4***	**0.56***	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	15-30	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	30-45	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
NOA-407854	0-15	**4.0**	**3.1**	**3.0**	**3.2**	**1.3**	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	15-30	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	30-45	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
NOA-447204	0-15	<0.50	<0.50	<0.50	**1.0**	**2.6**	**3.2**	**3.7**	**4.1**	**4.1**	**3.4**	**2.6**	**2.0**	**2.3**	**1.7**	**0.77**	**0.73**
	15-30	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	30-45	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
Cloquintocet-mexyl (CGA-185072)	0-15	**2.4**	**2.6**	**2.6**	**1.8**	**0.78**	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	15-30	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	30-45	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
CGA-153433	0-15	<0.50	<0.50	<0.50	<0.50	**0.57**	**0.52**	**0.58**	**0.58**	**0.85**	**0.56**	**0.62**	**0.81**	**1.3**	**0.82**	**0.52**	<0.50
	15-30	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
	30-45	NS	NS	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50

Data were btained from Tables 3a-3c, pp. 38-40 of the study report. Mean values are averages of three replicates. The method LOQ was 0.5 ppb for each analyte in soil. Total extractable and non-extractable residues and total recovery were not determined.

* Residue values above the limit of quantitiation were observed in only one or two of the three replicate samples. The average value was calculated by substituting 0.5 ppb for the replicate residue levels that were <0.5 ppb.

NS = no sample.

4. PARENT COMPOUND: In the bare ground plot, the measured zero-time concentration in the 0-15 cm soil depth was 22 ppb, which is 45% of the applied rate (reviewer-calculated based on a theoretical day-0 concentration of 0.049 mg a.i./kg in the 0-15 cm soil depth; Table 4a). The day-0 concentration of pinoxaden + NOA-407854 (after converting to parent equivalents) was 73% of the applied rate (reviewer-calculated). Following day 0, pinoxaden decreased to 7.5 ppb by 3 days, and was last detected above the LOQ at 1.3 ppb at 14 days posttreatment. Pinoxaden was not detected above the LOQ in soil below the 0-15 cm depth (Tables 4b-4c). The reviewer-calculated half-life of pinoxaden in soil under terrestrial field conditions was 3 days ($r^2 = 0.90$), based on all data points for the top 0-15 cm soil layer. The half-life value was calculated using linear regression analysis performed on a plot of ln-transformed concentrations vs. time and the equation $t_{½} = ln\ 2 / k$, where k is the rate constant. A DT90 value was not calculated. The dissipation pattern of pinoxaden was linear.

In the cropped plot, the maximum measured concentration in the 0-15 cm soil depth was 8.5 ppb at 0.5 days, which is 18% of the applied rate (reviewer-calculated based on a theoretical day-0 concentration of 0.048 mg a.i./kg in the 0-15 cm soil depth; Table 3a). The maximum concentration of pinoxaden + NOA-407854 (after converting to parent equivalents) was 27% of the applied rate at 0.5 days posttreatment (reviewer-calculated). Following 0.5 days, pinoxaden decreased to 4.1 ppb by 3 days, and was last detected above the LOQ at 0.56 ppb at 14 days posttreatment. Pinoxaden was not detected above the LOQ in soil below the 0-15 cm depth (Tables 3b-3c). The reviewer-calculated half-life of pinoxaden in soil under terrestrial field conditions was 4 days ($r^2 = 0.81$), based on all data points for the top 0-15 cm soil layer. The half-life value was calculated using linear regression analysis performed on a plot of ln-transformed concentrations vs. time and the equation $t_{½} = ln\ 2 / k$, where k is the rate constant. A DT90 value was not calculated. The dissipation pattern of pinoxaden was linear.

5. TRANSFORMATION PRODUCTS: After converting transformation product values to parent equivalents by dividing by the corresponding molecular weight conversion factors (0.79 for NOA-407854 and 0.82 for NOA-447204; see Reviewer Comment 1 for sample calculation), **NOA-407854** (7H-pyrazolo[1,2-d][1,4,5]oxadiazepine-7,9(8H)-dione, 8-(2,6-diethyl-4-methylphenyl)tetrahydro-) and **NOA-447204** (7H-pyrazolo[1,2-d][1,4,5]oxadiazepine-7,9(8H)-dione, 8-(2,6-diethyl-4-methylphenyl)tetrahydro-8-hydroxy-) were both detected in the soil at a concentration greater than 10% of the applied, based on the theoretical amount expected to be found in soil (Tables 3a-4c).

In the bare ground plot, **NOA-407854** was initially detected in the 0-15 cm soil depth at 11 ppb immediately following the application (which is 28.4% of the applied pinoxaden, after converting to parent equivalents; Table 4a). NOA-407854 increased to a maximum concentration of 14 ppb by 0.5 days (which is 36.2% of the applied pinoxaden, after converting to parent equivalents), then decreased to 6.1 ppb by 3 days, and was last detected above the LOQ at 0.74 ppb at 21 days posttreatment. NOA-407854 was not detected above the LOQ in soil below the 0-15 cm depth (Tables 4b-4c). The registrant-calculated half-life value for NOA-407854 was 3.3 days, calculated using non-linear regression (Figure 10). **NOA-447204** was initially detected in the 0-15 cm soil depth at 0.54 ppb at 0.5 days, increased to a maximum concentration of 4.2 ppb by 7 days (which is 10.5% of the applied pinoxaden, after converting to parent equivalents), then

decreased to 2.7 ppb by 125 days, and was 0.58 ppb at 494 days posttreatment. NOA-447204 was not detected above the LOQ in soil below the 0-15 cm depth.

In the cropped plot, **NOA-407854** was detected in the 0-15 cm soil depth at a maximum concentration of 4.0 ppb immediately following the application (which is 10.5% of the applied pinoxaden, after converting to parent equivalents), and decreased to a final concentration of 1.3 ppb by 7 days posttreatment (Table 3a). NOA-407854 was not detected above the LOQ in soil below the 0-15 cm depth (Tables 3b-3c). The registrant-calculated half-life value for NOA-407854 was 5.8 days, calculated using non-linear regression (Figure 4). **NOA-447204** was initially detected in the 0-15 cm soil depth at 1.0 ppb at 3 days, increased to a maximum concentration of 4.1 ppb by 35-45 days (which is 10.4% of the applied pinoxaden, after converting to parent equivalents), then decreased to 0.73 ppb by 494 days posttreatment. NOA-447204 was not detected above the LOQ in soil below the 0-15 cm depth.

Table 9: Chemical names and CAS numbers for the transformation products of pinoxaden.

Applicant's Code Name	CAS Number	CAS Chemical Name	Chemical formula	Molecular weight	SMILES string
NOA-407854	314020-44-5	7H-Pyrazolo[1,2-d][1,4,5] oxadiazepine-7,9(8H)-dione, 8-(2,6-diethyl-4-methylphenyl)tetrahydro-	$C_{18}H_{24}N_2O_3$	316.5	Not available
NOA-447204	Not available	7H-Pyrazolo[1,2-d][1,4,5] oxadiazepine-7,9(8H)-dione, 8-(2,6-diethyl-4-methylphenyl)tetrahydro-8-hydroxy-	$C_{18}H_{24}N_2O_4$	328.5	Not available

Data were obtained from p. 29 and Appendix 2, Figure 1, pp. 276-277 of the study report. The chemical formulas and molecular weight of NOA-447204 were determined by the reviewer based on the structures presented in Appendix 2.

6. EXTRACTABLE AND NON-EXTRACTABLE RESIDUES: Non-extractable residues were not measured.

Table 10: Dissipation routes of pinoxaden under field conditions.

Route of dissipation	% of applied amount (at the end of study period)	
	Bare ground plot	Cropped plot
Accumulation (residues) in soil/ carry over	0%	0%
Transformation (% of transformation products)	1.4%	1.9%
Leaching, if measured	Pinoxaden and its transformation products were not detected below the LOQ in soil below the 0-15 cm depth.	Pinoxaden and its transformation products were not detected below the LOQ in soil below the 0-15 cm depth.
Volatilization, if measured	Not measured	Not measured
Plant uptake, if measured	Not applicable	Not measured
Run off, if measured	Not measured	Not measured
Total		

Data were obtained from Tables 3a-4c, pp. 38-43 of the study report.

7. VOLATILIZATION: The concentration of applied pinoxaden lost through volatilization was not determined.

8. PLANT UPTAKE: Plant uptake was not determined.

9. LEACHING: No analytes were detected above the LOQ in soil below the 0-15 cm depth in either test plot (Tables 3a-4c).

10. RUN OFF: Run off was not studied.

11. RESIDUE CARRYOVER: DT90 values were not calculated. At the end of the study period, 494 days, 0% of the applied parent compound was detected in the bare ground and cropped test plots, and has no potential to carryover (Tables 3a-4c). At the end of the study period, carryover of the transformation products was 1.4-1.9% of the applied parent compound; the only transformation product detected was NOA-447204.

12. SUPPLEMENTARY STUDY RESULTS: Pinoxaden was not stable in frozen soil, with a mean corrected recovery of 100% at day 0 decreasing to 71% by 6 months and 42% by 12 months (MRID 46203022); recoveries were corrected for the mean procedural recovery. Transformation products NOA-407854 and NOA-447204 were both stable over time, with mean corrected recoveries ranging from 94-129% and 97-114%, respectively, with no pattern of decline.

III. STUDY DEFICIENCIES: Pinoxaden was not stable in frozen storage, decreasing to a corrected recovery of 55% following 9 months and 42% following 12 months (MRID 46203022). It is necessary to demonstrate the stability of the parent compound under typical storage conditions to ensure that the degradation of the parent occurred in the field and not during storage of the test samples. If pinoxaden was degrading during storage of the samples, then the calculated half-life value will not correctly reflect dissipation in the field.

IV. REVIEWER'S COMMENTS:

1. To determine percentage of the applied values for the transformation products, transformation product concentration values were converted to parent equivalents by dividing by the corresponding molecular weight conversion factors (0.79 for NOA-407854 and 0.82 for NOA-447204). Molecular weight conversion factors are calculated by dividing the molecular weight of the transformation product by the molecular weight of the parent. For example, for NOA-407854, 316.5 g/mol divided by 400.5 g/mol equals 0.79). To convert transformation product concentrations to parent equivalents, divide the transformation product concentration by the molecular weight conversion factor. For example, for NOA-407854, 11 ppb (detection at day 0, bare ground plot, 0-15 cm soil depth) divided by 0.79 equals 13.9 ppb parent equivalents.

   The percent of each transformation product in terms of percent of the applied pinoxaden was calculated by dividing the concentration of the transformation product in parent equivalents (see above on how to convert to parent equivalents) by the theoretical day-0 concentration of pinoxaden in the 0-15 cm soil depth (0.049 mg a.i./kg for the bare ground plot and 0.048 mg a.i./kg for the cropped plot; registrant-calculated; see footnote 1 to DER Table 5). For example, for NOA-407854, 13.9 ppb parent equivalents (detection at day 0, bare ground plot, 0-15 cm soil depth, see above for calculation) divided by 49 µg a.i./kg = 0.284 or 28.4% of the applied pinoxaden.

2. The registrant-calculated half-life of pinoxaden in the bare ground and cropped plots was 2.2 days ($r^2 = 0.98$) and 3.0 days ($r^2 = 0.97$), respectively, based on non-linear first-order analysis (SigmaPlot™ 8.0 for Windows) and determined using residues from the 0-15 cm soil depth (Figures 3-3a; and Figures 9-9a).

3. Mean procedural recoveries from soil samples that were fortified with pinoxaden, NOA-407854, and NOA-447204 at 0.5, 5.0, and 50 ppb and analyzed concurrently with samples from the bare ground plot were 94 ± 12.8%, 80 ± 12.4%, and 80 ± 12.9%, respectively (Appendix 2, Tables 12-13). For the cropped plot, mean procedural recoveries were 93 ± 13.4% for pinoxaden, 78 ± 11.7% for NOA-407854, and 79 ± 11.6% for NOA-447204 (Appendix 2, Tables 5-6).
4. Test samples from the treated bare ground and cropped plots were analyzed for residues of the safener, cloquintocet-mexyl and its transformation product CGA-153433 (acetic acid, [(5-chloro-8-quinolinyl)oxy]-; Appendix 2, Figure 1). The results are included in DER Tables 7 and 8. All other analytical results for the safener and its transformation product are reported

below. The mean recoveries of cloquintocet-mexyl from the filter papers placed in the treated bare ground and cropped plots were 88% for the bare ground plot and 62% for the cropped plot (Table 2). The mean concentrations of cloquintocet-mexyl from the tank mix samples collected prior to and following the application ranged from 121-123% for the bare ground plot and 82-86% for the cropped plot (Table 1). Mean recoveries of cloquintocet-mexyl and CGA-153433 from method validation samples fortified with each analyte at 0.50, 5.0, and 50 ppb were 90 ± 5.3% for cloquintocet-mexyl and 97 ± 5.6% for CGA-153433 (mean recoveries are across all three fortification levels; Appendix 2, Table 1). Mean procedural recoveries from soil samples fortified at 0.5, 5.0, and 50 ppb from the bare ground plot were 82 ± 12.5% for cloquintocet-mexyl and 97 ± 15.6% for CGA-153433, and mean recoveries from the cropped plot were 79 ± 11.6% for cloquintocet-mexyl and 88 ± 14.8% for CGA-153433 (Appendix 2, and Tables 5-6 and 12-13). Mean recoveries from field spikes prepared for cloquintocet-mexyl and CGA-153433 by fortifying control soil samples at 10 ppb were 74% for cloquintocet-mexyl and 86% for CGA-153433 (Table 5). Recoveries of cloquintocet-mexyl and CGA-153433 from soil samples that were fortified at 5 ppb and stored frozen for up to 12 months ranged from 91-135% and 95-112%, respectively, indicating stability during storage (recoveries were corrected for concurrent recoveries; MRID 46203022).

5. The laboratory storage stability study was not conducted for a length of time equal to or exceeding the longest storage interval for the test samples. The interim report for the storage stability study reported recoveries following up to 12 months, whereas the test samples from the field dissipation site were stored for up to 442-449 days (not including samples that were re-analyzed). The reviewer notes that the storage stability study report was an interim report and that additional samples were scheduled to be collected following 15 and 18 months of storage.

V. REFERENCES:

1. U.S. Environmental Protection Agency. 1982. Pesticide Assessment Guidelines, Subdivision N, Chemistry: Environmental Fate, Section 164-1, Terrestrial Field Dissipation Studies. Office of Pesticide and Toxic Substances, Washington, DC. EPA 540/9-82-021.

2. U.S. Environmental Protection Agency. 1993. Pesticide Registration Rejection Rate Analysis - Environmental Fate. Office of the Prevention, Pesticides, and Toxic Substances, Washington, DC. EPA 738-R-93-010.

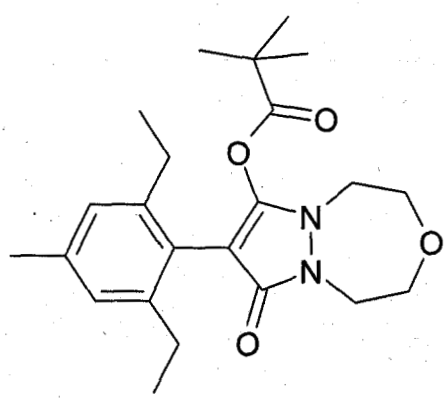
3. U.S. Environmental Protection Agency. 1989. FIFRA Accelerated Reregistration, Phase 3 Technical Guidance. Office of the Prevention, Pesticides, and Toxic Substances, Washington, DC. EPA 540/09-90-078.

Attachment 1

Structures of Parent and Transformation Products

Pinoxaden [NOA-407855]

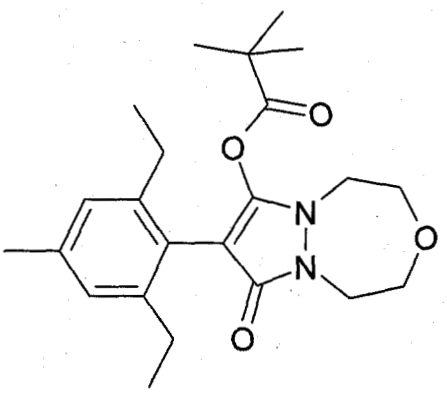
IUPAC name:	8-(2,6-Diethyl-p-tolyl)-1,2,4,5-tetrahydro-7-oxo-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-9-yl 2,2-dimethylpropionate.
CAS name:	8-(2,6-Diethyl-4-methylphenyl)-1,2,4,5-tetrahydro-7-oxo-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-9-yl 2,2-dimethylpropanoate. 8-(2,6-Diethyl-4-methylphenyl)-1,2,4,5-tetrahydro-7-oxo-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-9-yl ester 2,2-dimethylpropanoic acid.
CAS No:	243973-20-8.
SMILES string:	O=C1C(=C(N2N1CCOCC2)OC(=O)C(C)(C)C)c1c(cc(cc1CC)C)CC.



Identified Compounds

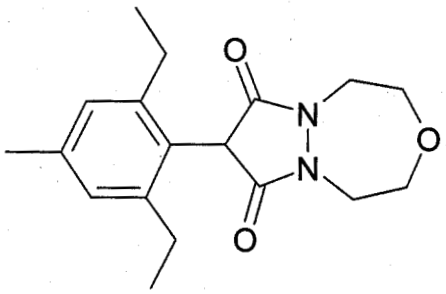
Pinoxaden [NOA-407855]

IUPAC name:	8-(2,6-Diethyl-p-tolyl)-1,2,4,5-tetrahydro-7-oxo-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-9-yl 2,2-dimethylpropionate.
CAS name:	8-(2,6-Diethyl-4-methylphenyl)-1,2,4,5-tetrahydro-7-oxo-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-9-yl 2,2-dimethylpropanoate. 8-(2,6-Diethyl-4-methylphenyl)-1,2,4,5-tetrahydro-7-oxo-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-9-yl ester 2,2-dimethylpropanoic acid.
CAS No:	243973-20-8.
SMILES string:	O=C1C(=C(N2N1CCOCC2)OC(=O)C(C)(C)C)c1c(cc(cc1CC)C)CC.

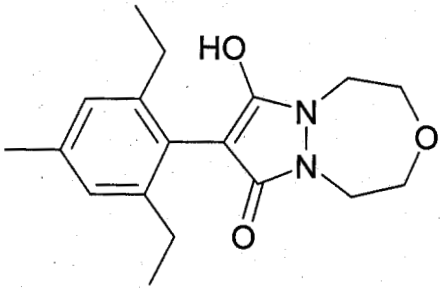


NOA-407854

IUPAC name:	8-(2,6-Diethyl-4-methyl-phenyl)tetrahydro-pyrazolo[1,2-d][1,4,5]oxadiazepin-7,9-dione.
CAS name:	8-(2,6-Diethyl-4-methylphenyl)tetrahydro-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-7,9(8H)-dione. 8-(2,6-Diethyl-4-methylphenyl)-1,2,4,5-tetrahydro-9-hydroxy-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-7-one.
CAS No:	314020-44-5 (keto form); 243973-19-5 (enol form).



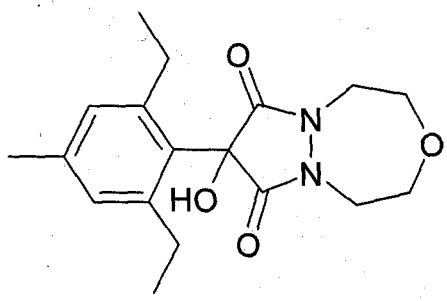
Keto form



Enol form

NOA-447204

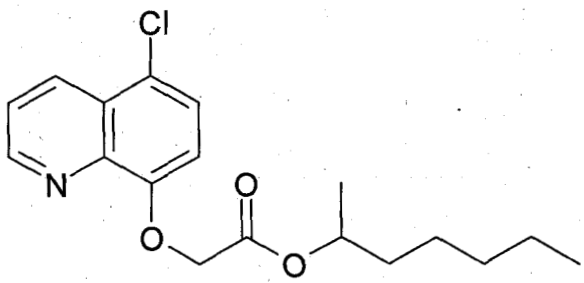
IUPAC name:	8-(2,6-Diethyl-4-methyl-phenyl)-8-hydroxy-tetrahydro-pyrazolo[1,2-d][1,4,5]oxadiazepin-7,9-dione.
CAS name:	8-(2,6-Diethyl-4-methylphenyl)tetrahydro-8-hydroxy-7H-pyrazolo[1,2-d][1,4,5]oxadiazepin-7,9(8H)-dione.
CAS No:	Not reported.
SMILES string:	O=C1C(c2c(cc(cc2CC)C)CC)(C(=O)N2N1CCOCC2)O.



Structure of the Safener and its Transformation Product

CGA-185072 [Safener; Cloquintocet-mexyl]

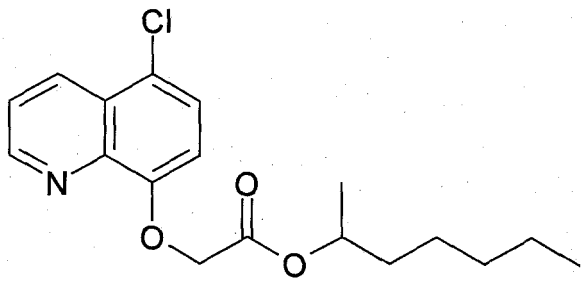
IUPAC name:	5-Chloro-8-quinolinoxyacetic acid-1-methylhexylester. (Previously known in structure files; see Safener of Clodinfop-propargyl)
CAS name:	1-Methylhexyl ester [(5-chloro-8-quinolinyl)oxy]-acetic acid.
CAS No:	99607-70-2.
SMILES string:	Clc1ccc(c2c1cccn2)OCC(=O)OC(CCCCC)C.



Identified Compounds

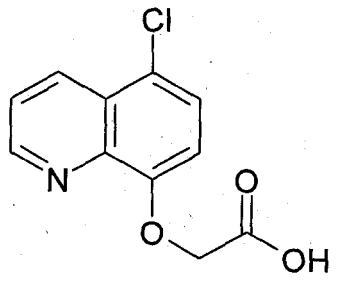
CGA-185072 [Safener; Cloquintocet-mexyl]

IUPAC name: 5-Chloro-8-quinolinoxyacetic acid-1-methylhexylester. (Previously known in structure files; see Safener of Clodinfop-propargyl)
CAS name: 1-Methylhexyl ester [(5-chloro-8-quinolinyl)oxy]-acetic acid.
CAS No: 99607-70-2.
SMILES string: Clc1ccc(c2c1cccn2)OCC(=O)OC(CCCCC)C.



CGA 153433

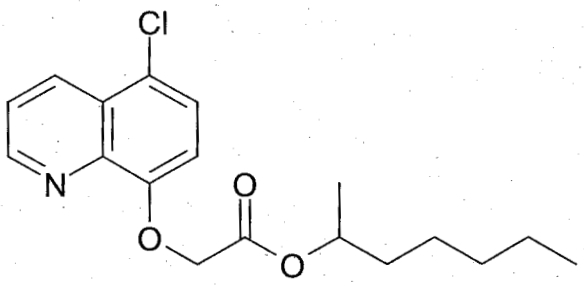
IUPAC name: Not reported.
CAS name: [(5-Chloro-8-quinolinyl)oxy]-acetic acid.
CAS No: 88349-88-6.
SMILES string: Clc1ccc(c2c1cccn2)OCC(=O)O.



Structure of the Safener and its Transformation Product

CGA-185072 [Safener; Cloquintocet-mexyl]

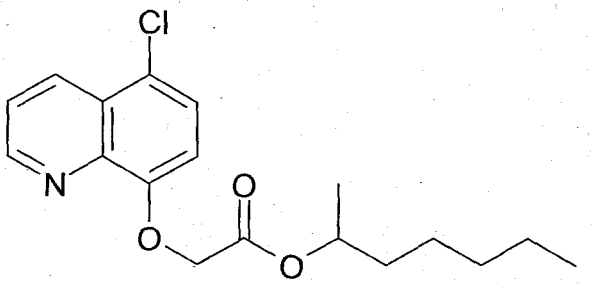
IUPAC name:	5-Chloro-8-quinolinoxyacetic acid-1-methylhexylester. (Previously known in structure files; see Safener of Clodinfop-propargyl)
CAS name:	1-Methylhexyl ester [(5-chloro-8-quinolinyl)oxy]-acetic acid.
CAS No:	99607-70-2.
SMILES string:	Clc1ccc(c2c1cccn2)OCC(=O)OC(CCCCC)C.



Identified Compounds

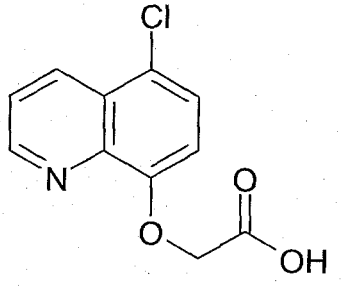
CGA-185072 [Safener; Cloquintocet-mexyl]

IUPAC name: 5-Chloro-8-quinolinoxyacetic acid-1-methylhexylester. (Previously known in structure files; see Safener of Clodinfop-propargyl)
CAS name: 1-Methylhexyl ester [(5-chloro-8-quinolinyl)oxy]-acetic acid.
CAS No: 99607-70-2.
SMILES string: Clc1ccc(c2c1cccn2)OCC(=O)OC(CCCCC)C.



CGA 153433

IUPAC name: Not reported.
CAS name: [(5-Chloro-8-quinolinyl)oxy]-acetic acid.
CAS No: 88349-88-6.
SMILES string: Clc1ccc(c2c1cccn2)OCC(=O)O.



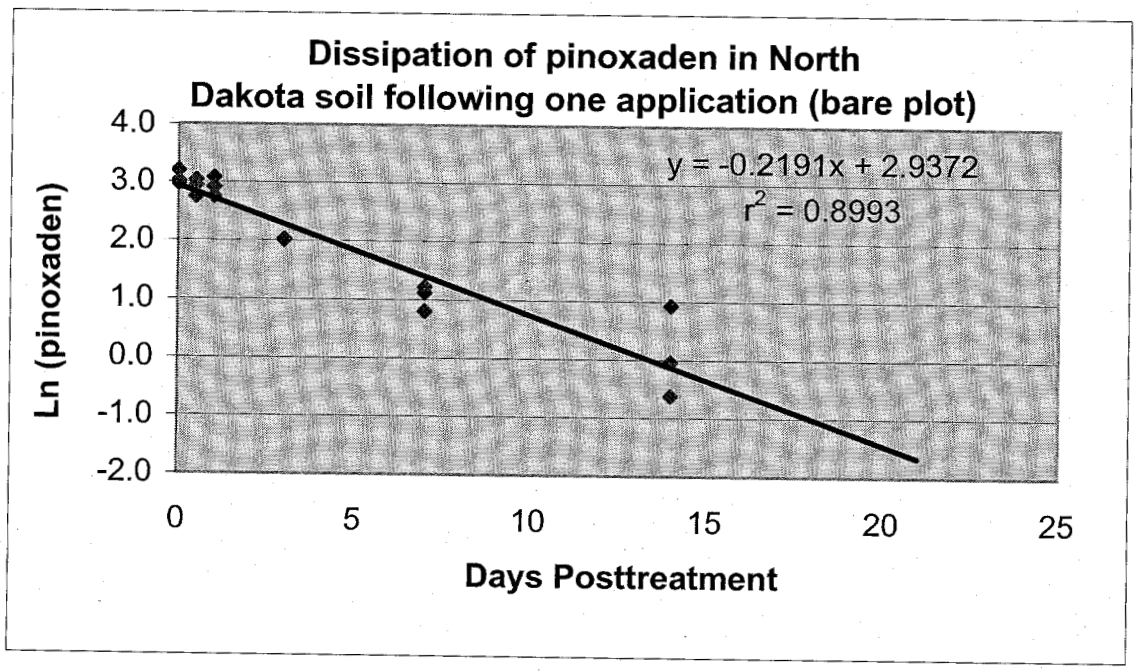
Attachment 2

Excel Spreadsheets

Chemical Name	Pinoxaden	**Bare-ground plot**
PC Code	147500	
MRID	46203023	**Half-life (days) = 3**
Guideline No.	164-1	**Half-life calculated using all replicate data**

Days Posttreatment	Pinoxaden (ppb)	Ln (pinoxaden)
0	20.4	3.0155
0	19.6	2.9755
0	24.5	3.1987
0.5	15.6	2.7473
0.5	19.0	2.9444
0.5	20.8	3.0350
1	15.7	2.7537
1	18.4	2.9124
1	21.7	3.0773
3	7.52	2.0176
3	7.56	2.0229
3	7.46	2.0096
7	2.22	0.7975
7	3.37	1.2149
7	3.06	1.1184
14	0.54	-0.6218
14	0.96	-0.0387
14	2.53	0.9282
21	<0.50	
21	<0.50	
21	<0.50	

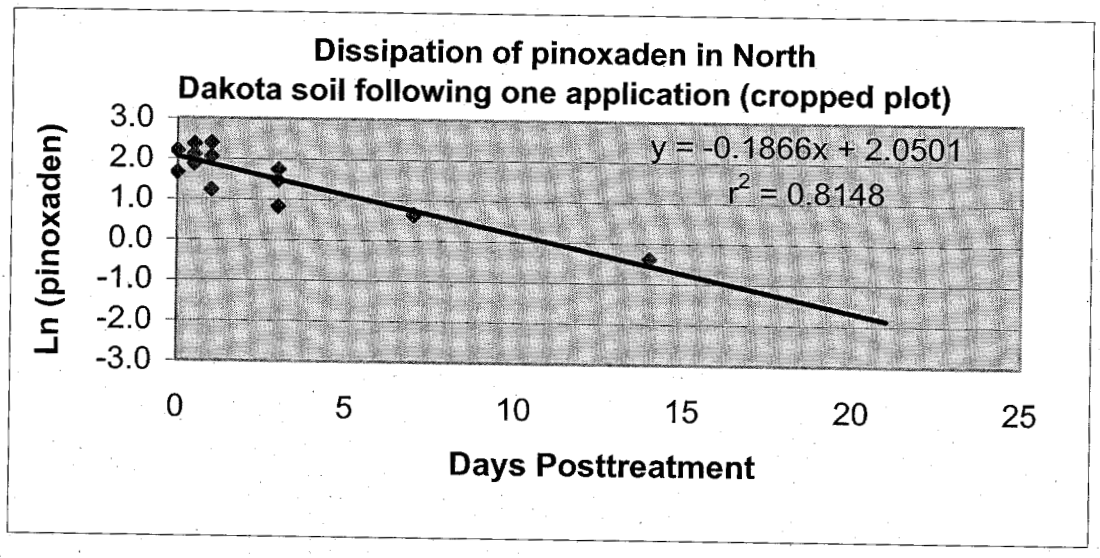
Data obtained from Appendix 2, Table 9, pp. 240-242 in the study report.
* Replicate values are reviewer-calculated means of multiple analyses where replicates were re-analyzed.



Chemical Name	Pinoxaden	**Cropped plot**
PC Code	147500	
MRID	46203023	**Half-life (days) = 4**
Guideline No.	164-1	**Half-life calculated using all replicate data**

Days Posttreatment	Pinoxaden (ppb)	Ln (pinoxaden)
0	8.96	2.1928
0	9.06	2.2039
0	5.23	1.6544
0.5	8.22	2.1066
0.5	6.51	1.8733
0.5	10.7	2.3702
1	10.9	2.3888
1	7.80	2.0541
1	3.43	1.2326
3	4.33	1.4656
3	5.70	1.7405
3	2.29	0.8286
7	1.87	0.6259
7	1.94	0.6627
7	<0.50	
14	0.675	-0.3930
14	<0.5	
14	<0.5	
21	<0.5	
21	<0.5	
21	<0.5	

Data obtained from Appendix 2, Table 2, pp. 218-219 in the study report.
* Replicate values are reviewer-calculated means of multiple analyses where replicates were re-analyzed.



Pages 41-59 *Access to FIFRA health and safety data is restricted under FIFRA section 10(g)*